CORNING

Micro Reactor Technology:
A Safer and Intelligent Approach to Process Intensification

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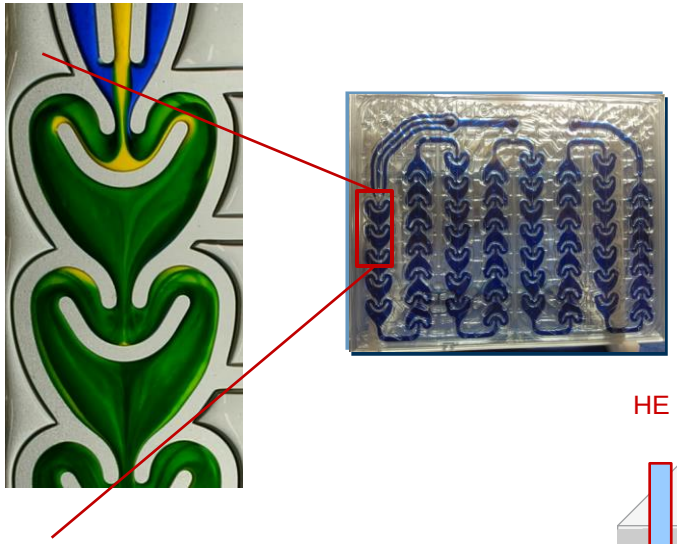
Agenda

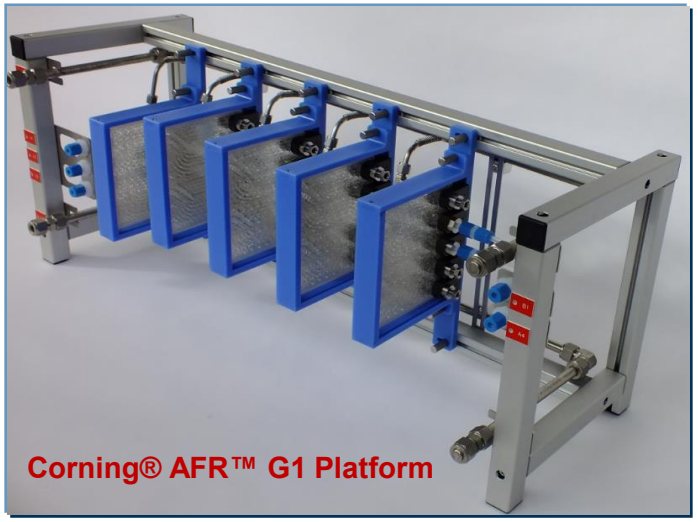
- ❑ **AFR™ Technology & Products**
- ❑ **AFR™-Production Made Real**
- ❑ **Concluding Remarks**

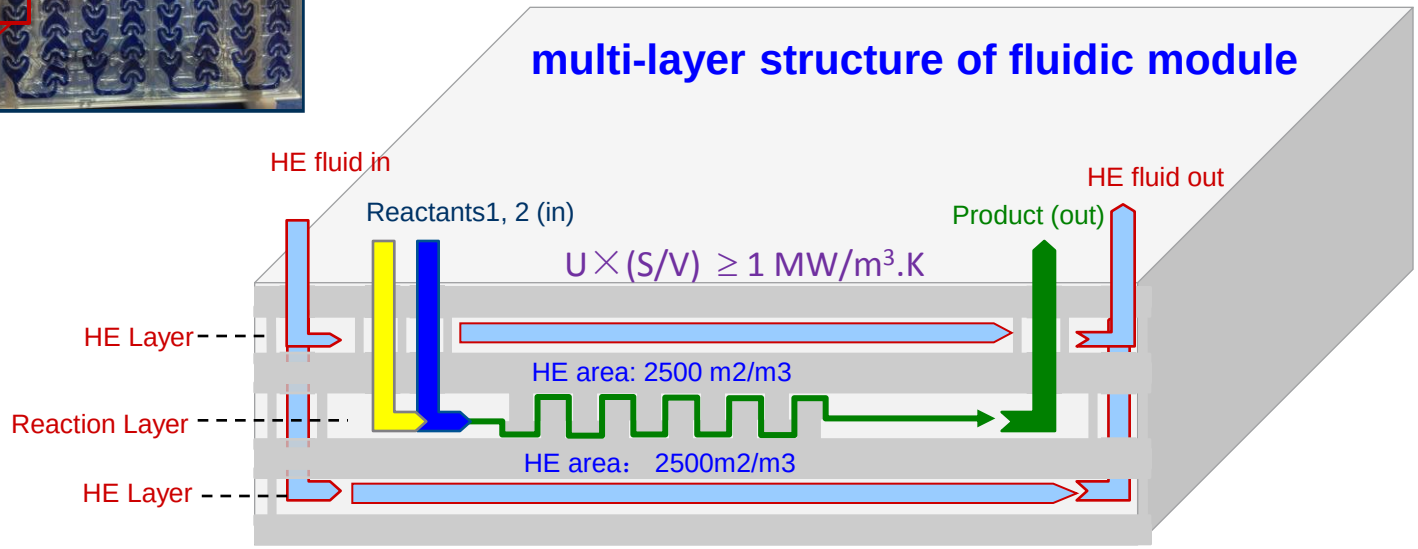
Corning Patented “Heart-Cell” Fluid Module Designs

Offer excellent mixing (100X better), superior heat transfer (1000X enhancement)

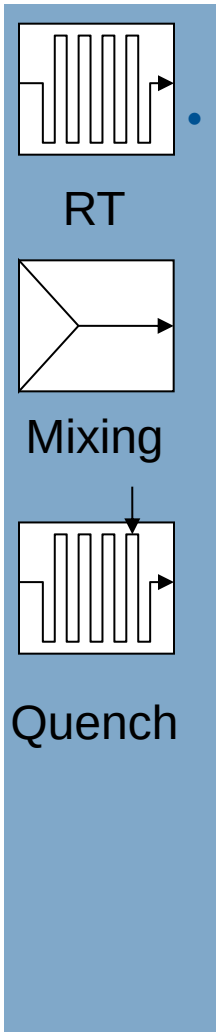
Mixing of Two Liquid Phases
Liquid 1: Blue
Liquid 2: Yellow
Mixture: Green





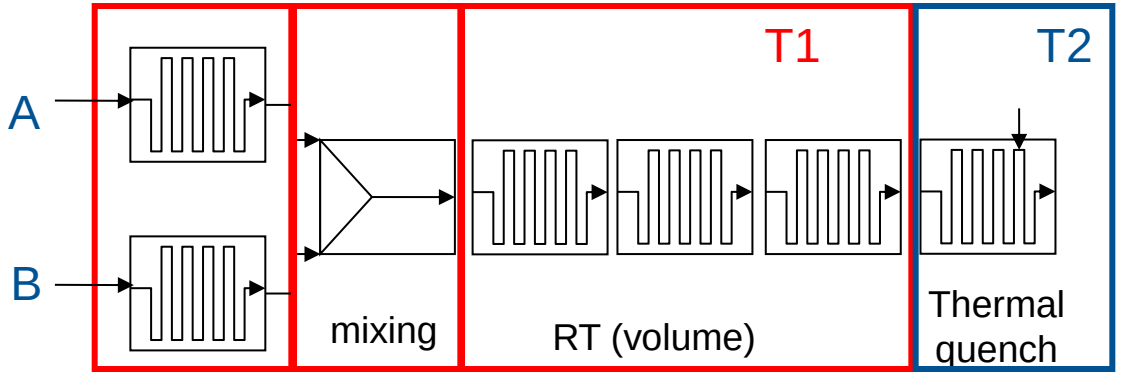


AFR Offers Process Config Flexibility & Multipurpose



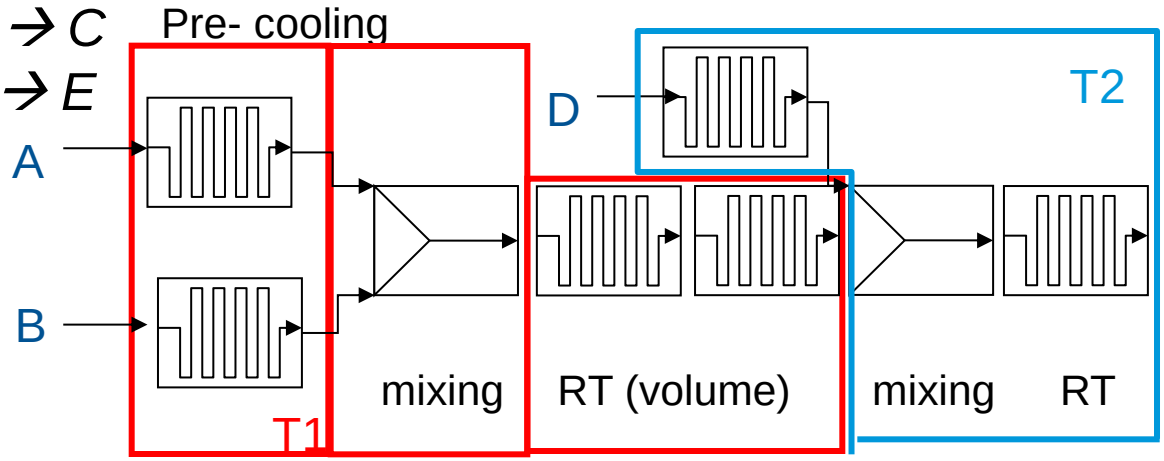
Flow reactors: A combination of individual steps

$A + B \rightarrow C$



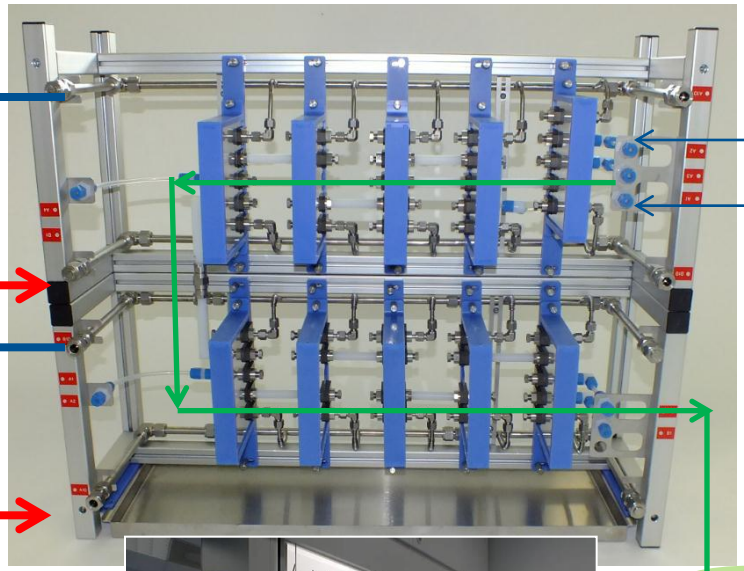
$A + B \rightarrow C$

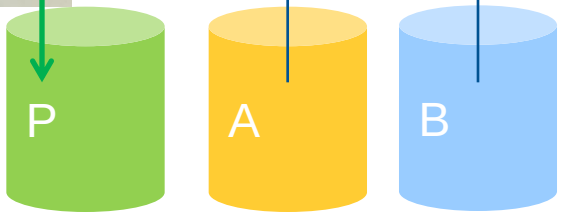
$C + D \rightarrow E$



Corning AFR™ G1 Process Development System Platform

Chiller for temperature Control: -20 to 200°C, 2.5 bar	**Corning AFR™ Reactor Platform** Operating pressure up to 18 bar	**Dosing Lines** (20 bar) (S.S. or metal-free pump-heads)

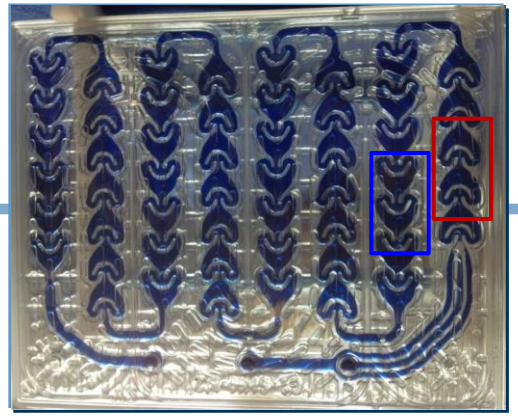




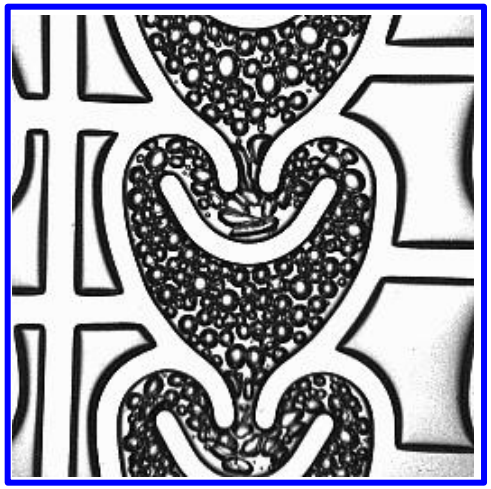
Gas-Liquid Mixing in AFR: *100X improvement of GL interfacial area*

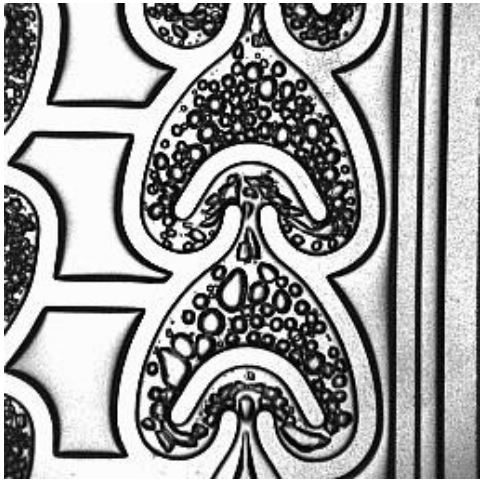
Up-flow heart-cell unit

L：50 ml/min
G：100 NmL/min

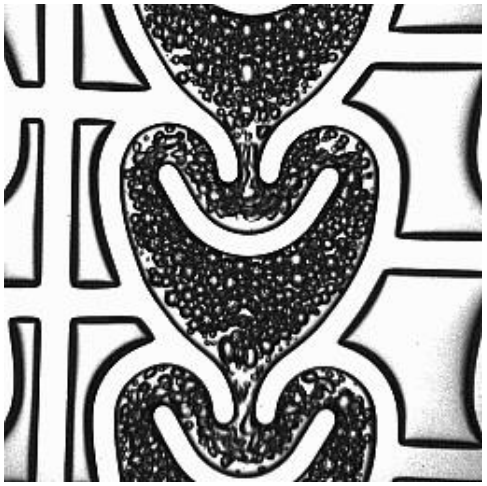


Down flow heart-cell unit

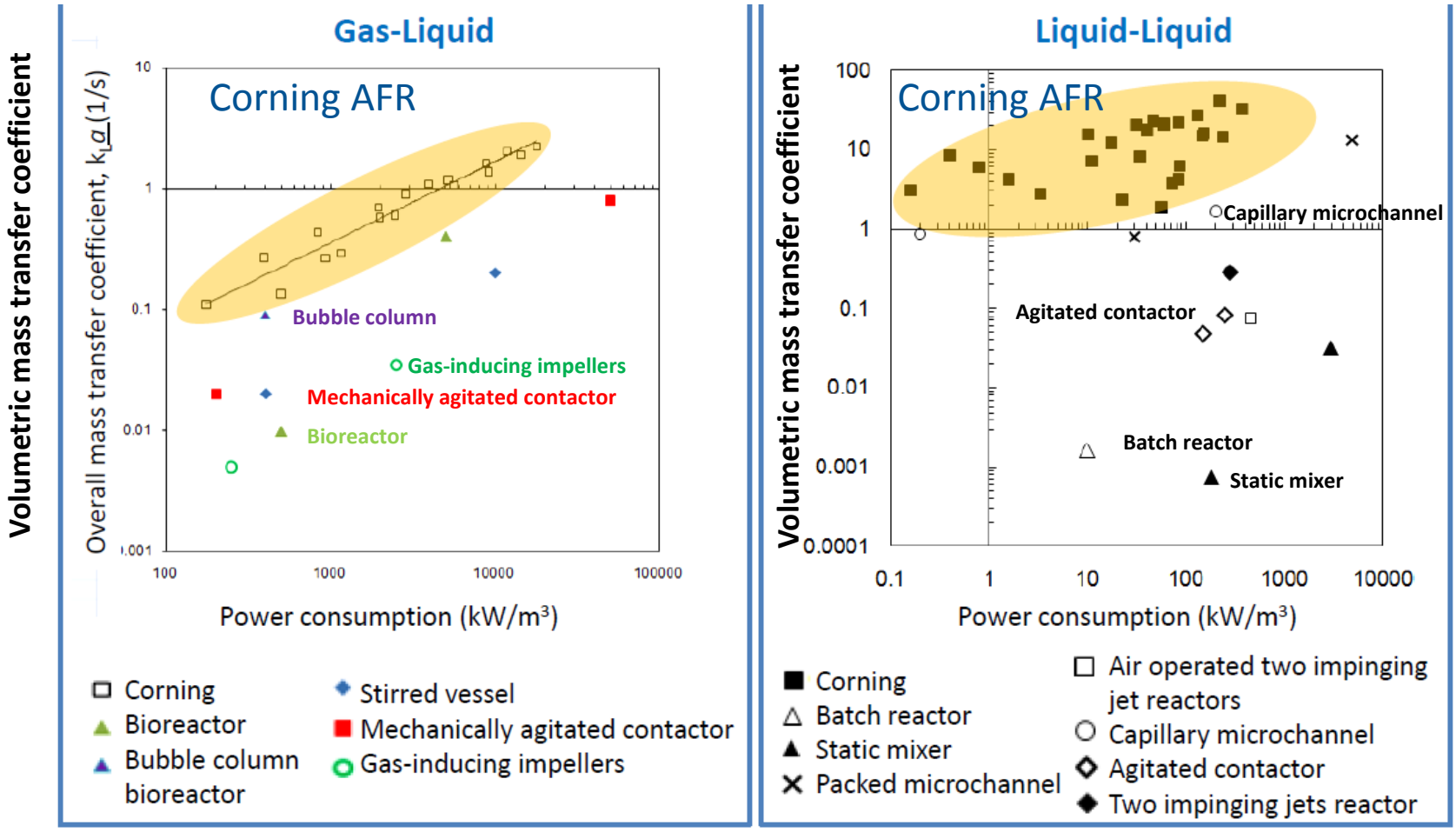




L：100 ml/min
G：200 NmL/min

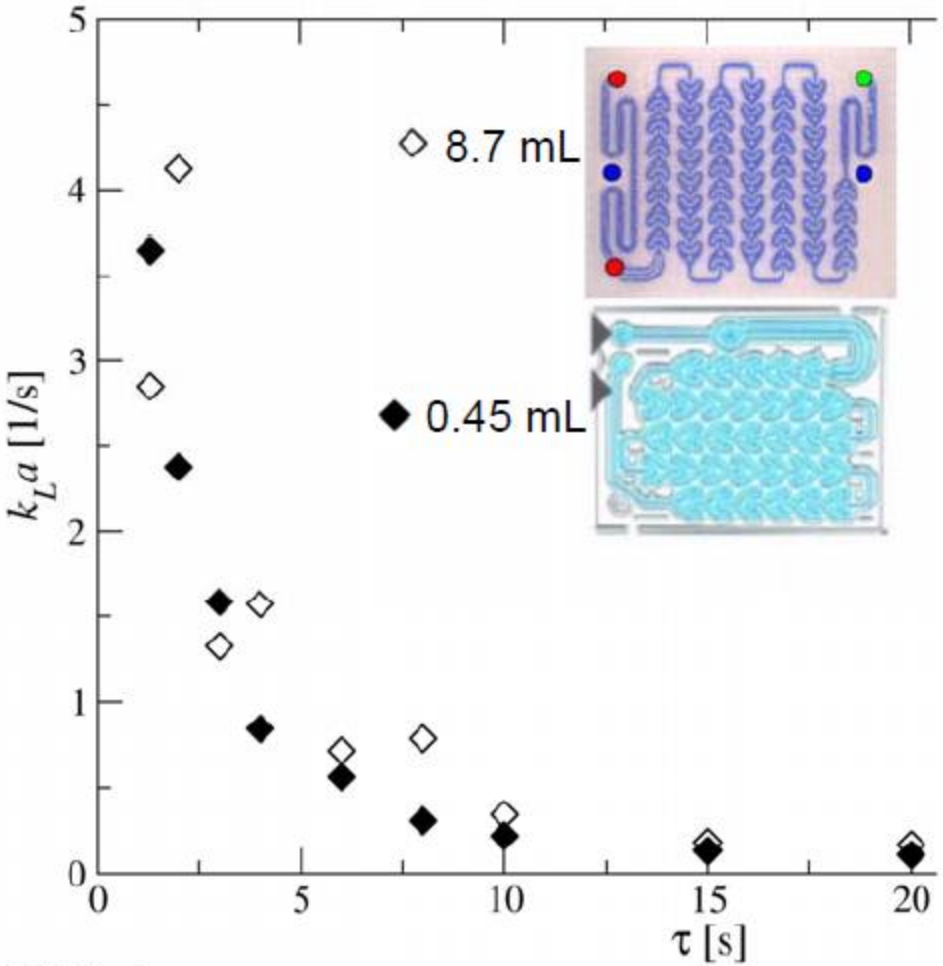


AFR™ Offers Superior G-L, L-L Mass Transfer Performance with Much Lower Energy Consumption (or Pressure-Drops)



- María José Nieves-Remacha,† Amol A. Kulkarni,†,‡ and Klavs F. Jensen; "**Gas−Liquid Flow** and Mass Transfer in an Advanced-Flow Reactor", *Ind. Eng. Chem. Res. 2013, 52, 8996−9010*
- María José Nieves-Remacha,† Amol A. Kulkarni,‡ and Klavs F. Jensen:" Hydrodynamics of **Liquid−Liquid Dispersion** in an Advanced-Flow Reactor", *Ind. Eng. Chem. Res. 2012, 51(50), 16251-16262*

Butanol-water: mass transfer in Corning AFR and LFR modules



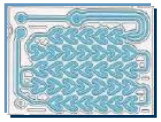
M. J. Nieves Remacha A. A. Kulkarni

- **Splitting and combining droplets provide effective mass transfer over different length scales**

S. Kuhn, A. Woitalka, K. F. Jensen, Scalability of mass transfer in liquid–liquid flow, *Chem Eng. Sci.*, 116 (2014) 1-8

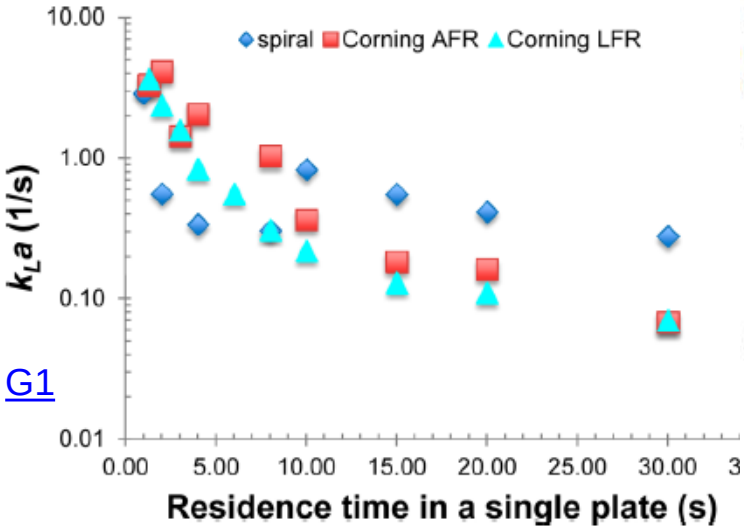
Scaleup: Oxidation production rate increases up to 700 times from spiral microreactor (MIT) to Corning G1 AFR

spiral



LFR

G1



Scheme 1. Optimized reaction conditions for the oxidation[34] of (a) 1-phenylethanol, (b) 3-nitrobenzyl alcohol, and (c) benzaldehyde[33]

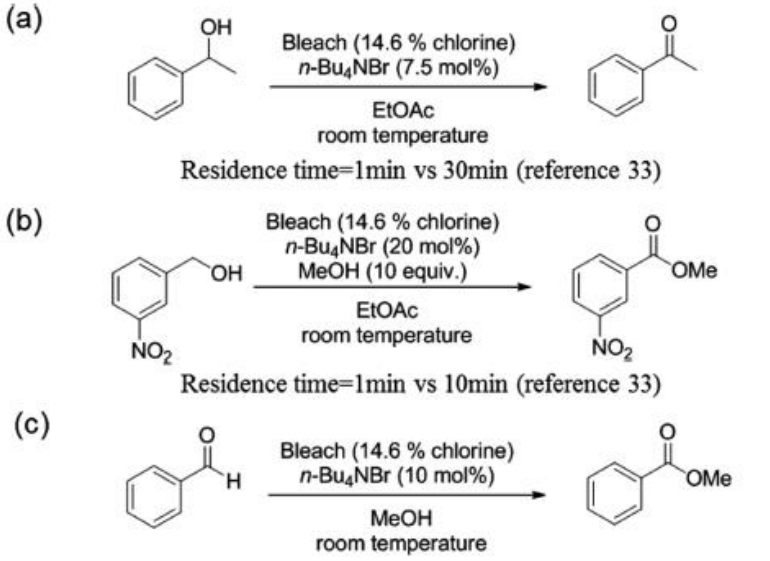
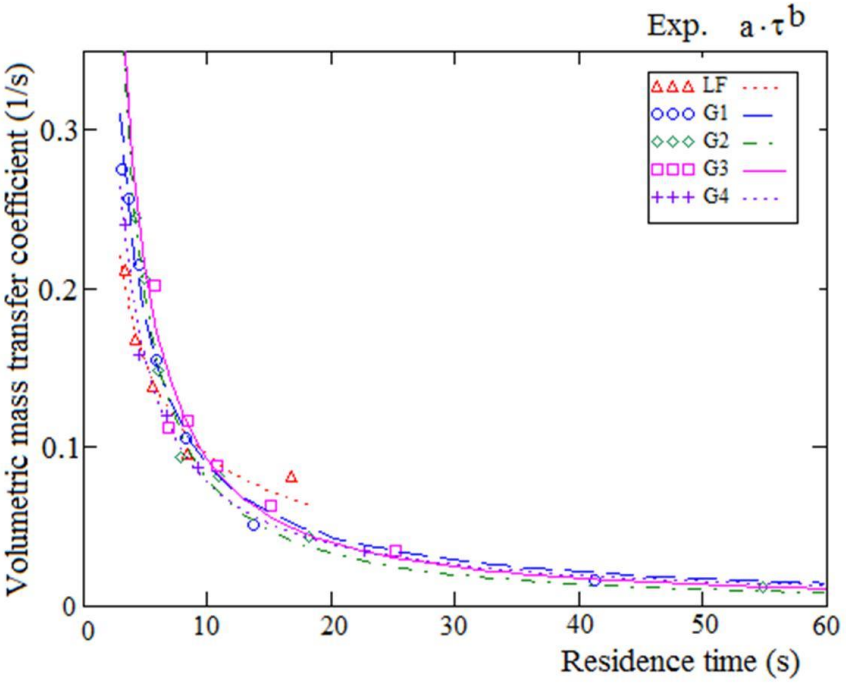


Table 1. Production rate comparison for oxidation reactions among different reactors

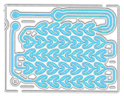
product	reactor type	production rate (g/min)
acetophenone	microreactor	0.0064
	Corning LFR	0.37
	Corning AFR	4.08
methyl 3-nitrobenzoate	microreactor	0.0063
	Corning LFR	0.16
	Corning AFR	1.76
methyl benzoate	microreactor	0.0027
	Corning LFR	0.075
	Corning AFR	0.85

Yanjie Zhang, Stephen C. Born, and Klavs F. Jense; Scale-Up Investigation of the Continuous Phase-Transfer-Catalyzed Hypochlorite Oxidation of Alcohols and Aldehydes; *Org. Process Res. Dev. 2014, 18, 1476−1481*

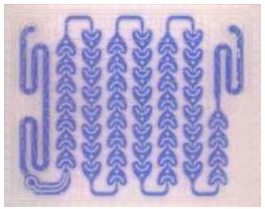
Consistent mixing and mass transfer performance from lab to production at same residence time



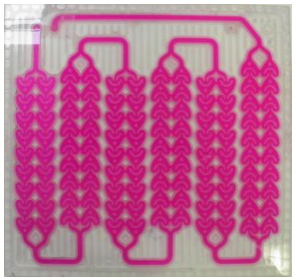
LFR 5 t/y



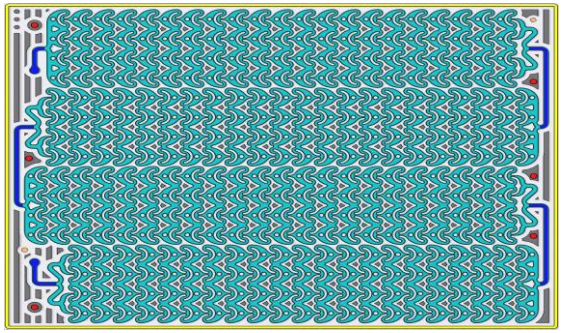
G1, 80 t/y



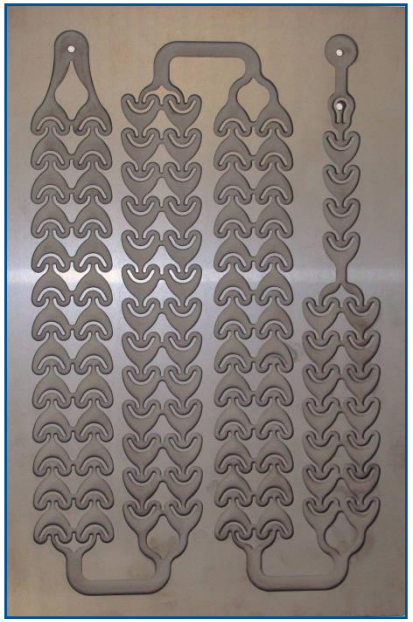
G2, 250 t/y



G3, 1000 t/y



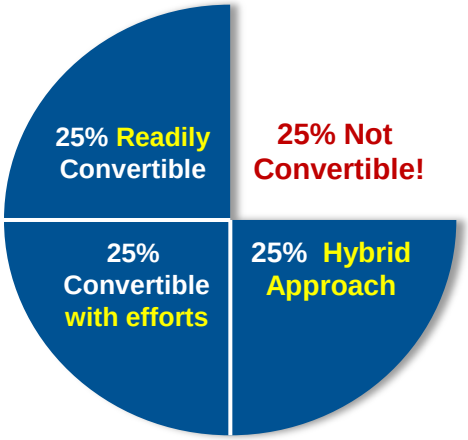
G4, 2000 t/y



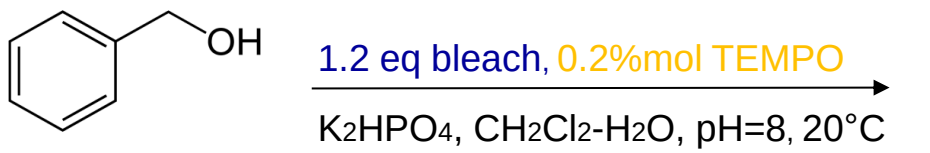
Chemistry Types Applicable in AFR

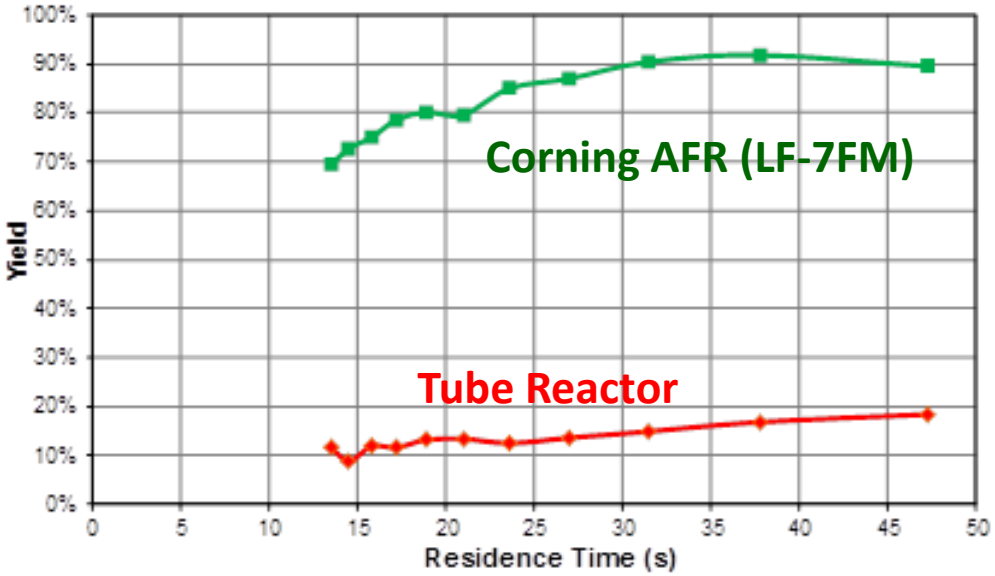
Halogenation	Chlorination
	Bromination
	Fluorination
	Hydrogenation
Low temperatures Friedel–Crafts	De-hydrogen
	Halogen exchange
	Friedel–Crafts acylation
	Friedel–Crafts Alkylation
Hydrogenation	Reduction of nitro group
	Reduction of doule bound
	Reduction of triple bound
	Reduction of chloro group
	De protection
Nitration	Nitration of alcohol
	Nitration of amine
	Nitration of Aromatic compounds
	Nitrosation of alcohol
Oxidation	Oxide double bond to alcohol
	Oxide methene to carbonyl group
	Oxide phenol to quinone
	oxide acid or alcohol to peroxide
Others	Suzuki coupling
	Hofmann degradation
	Hofmann rearrangement
	To form diazomethane
	To form diazo salt
	Grignard

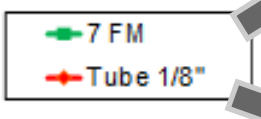
How many chemistry reactions can be converted from batch process to continuous AFR

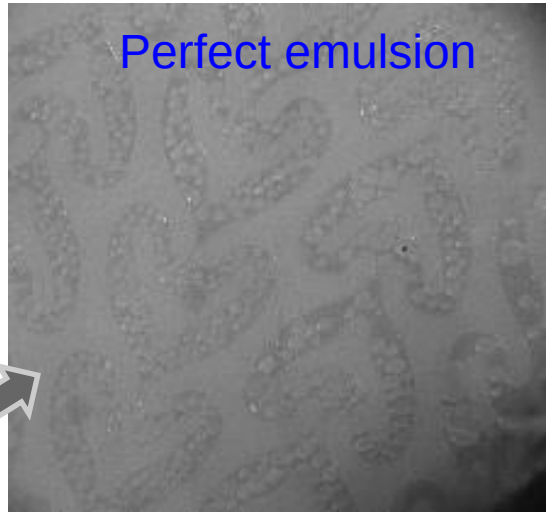


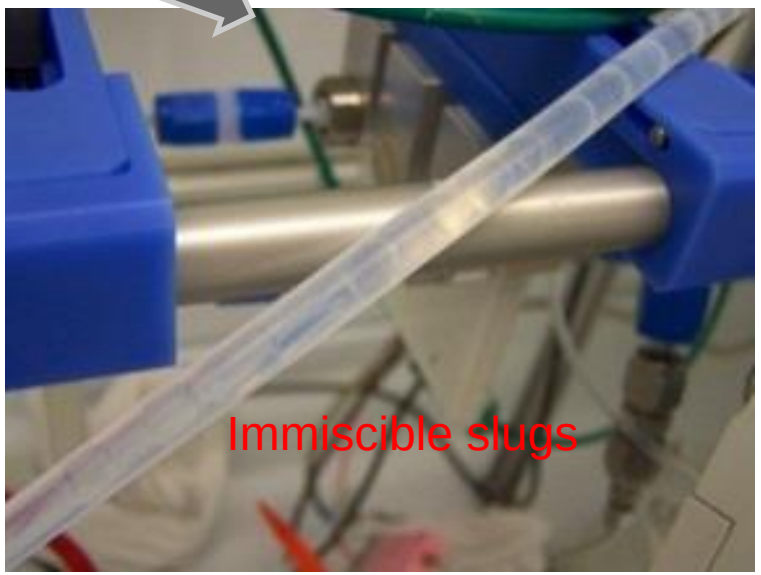
Corning AFR vs. Tube Reactor:
Excellent mixing matters







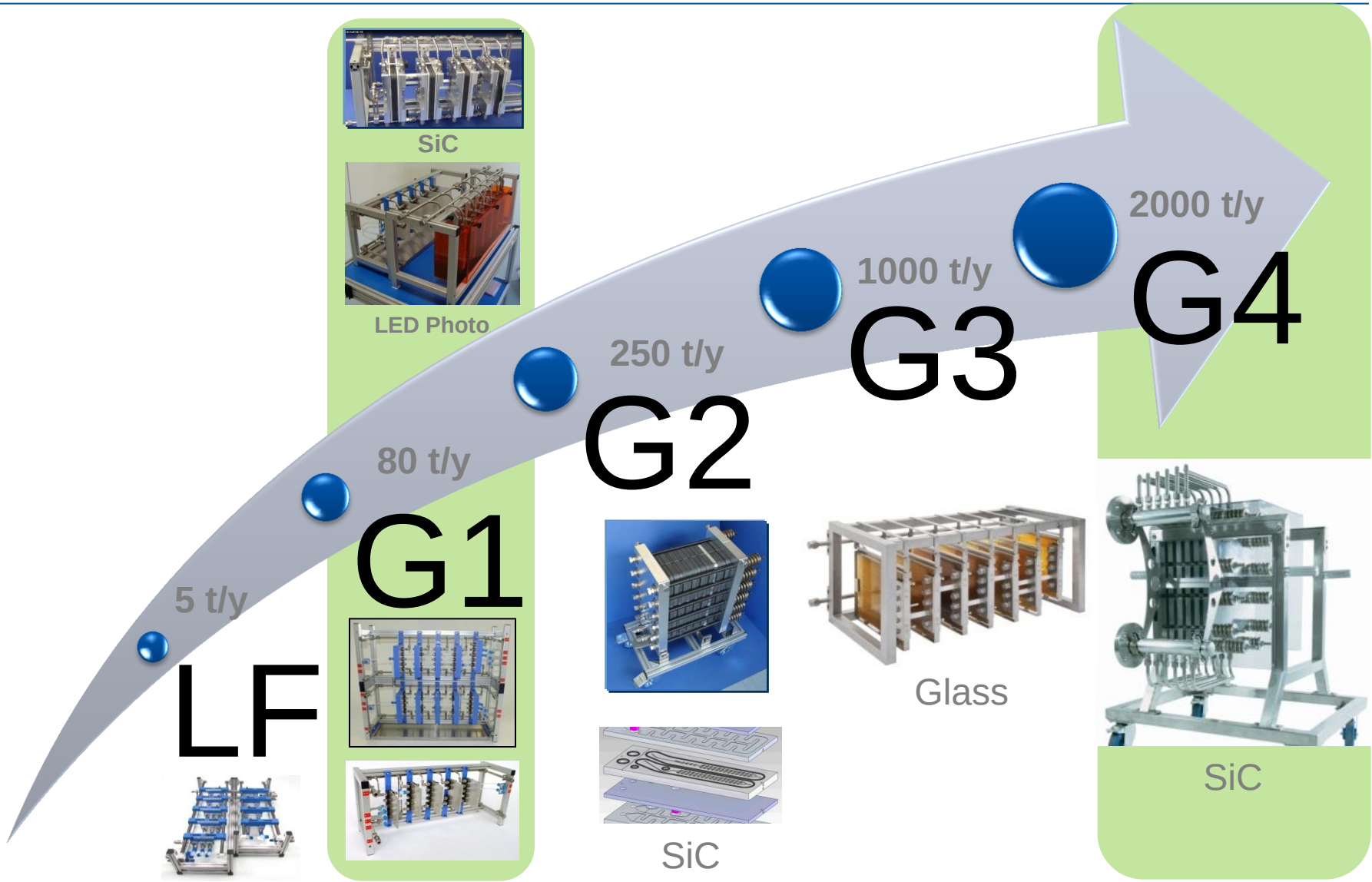




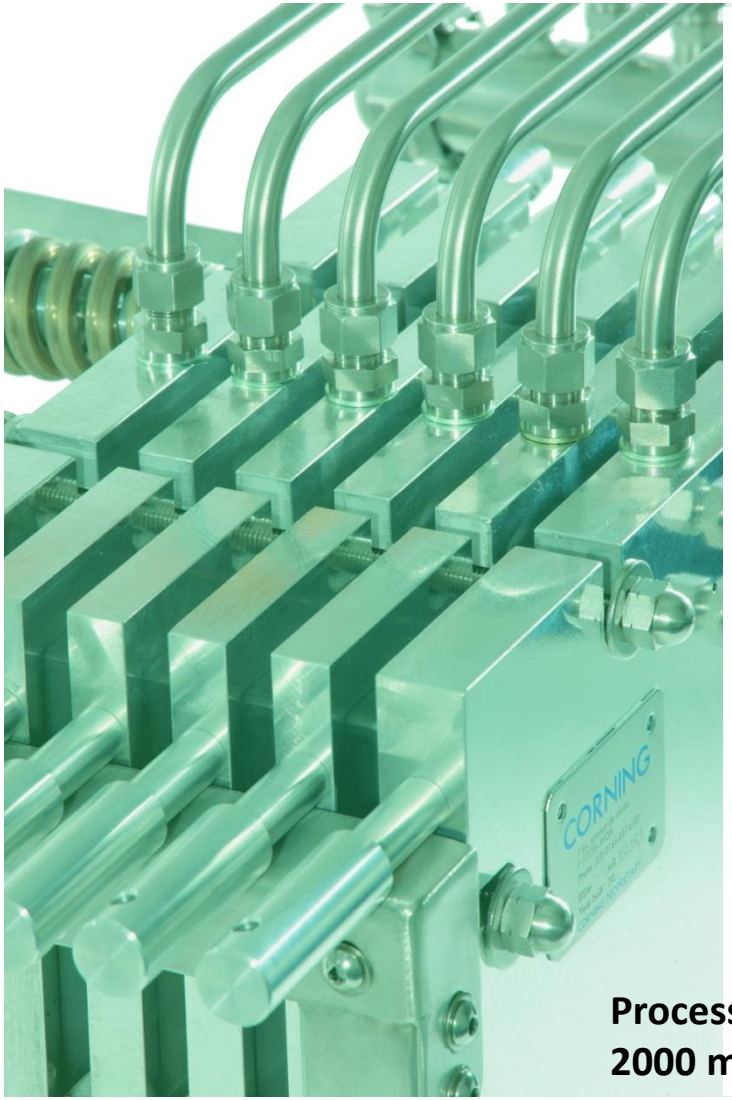
Organic-Aqueous immiscible mixture phases

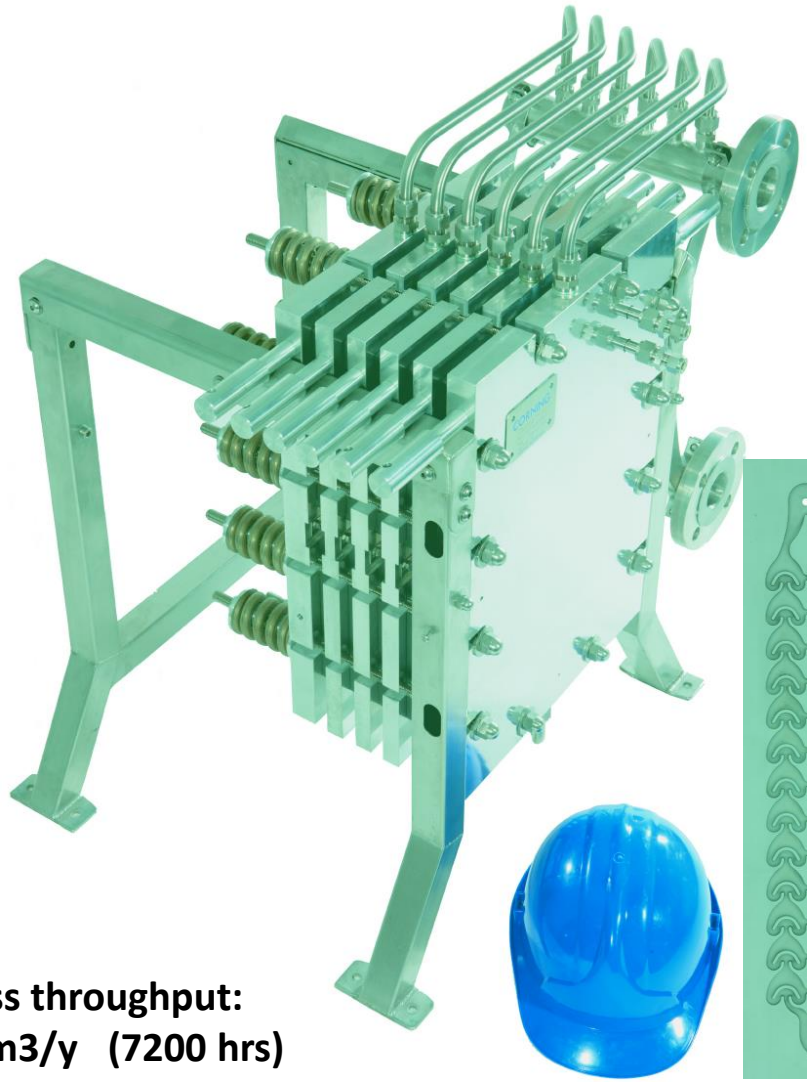
Continuous Innovations Offer Comprehensive Solutions

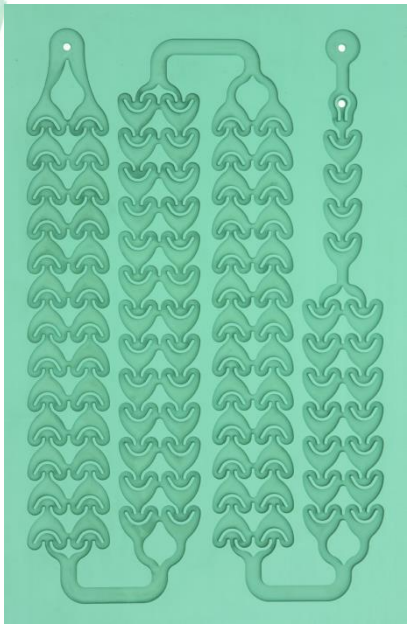
-Seamless scaleup from lab to manufacturing production



Corning G 4 Ceramic Reactor super-throughput providing competitive production performance



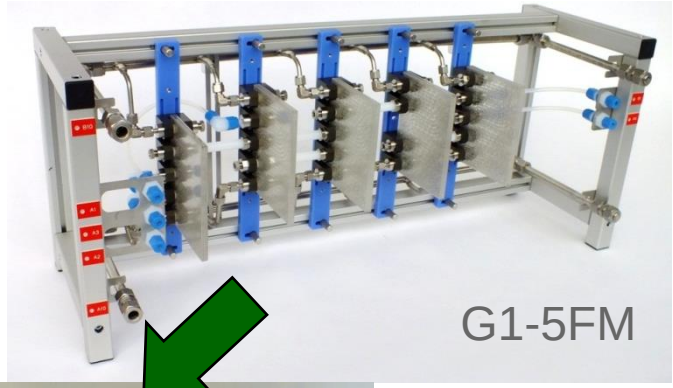


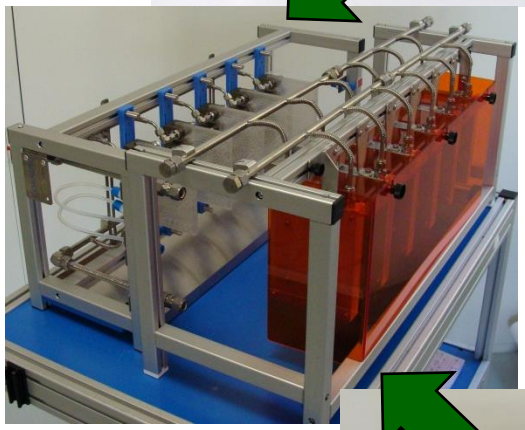


Process throughput:
2000 m3/y (7200 hrs)

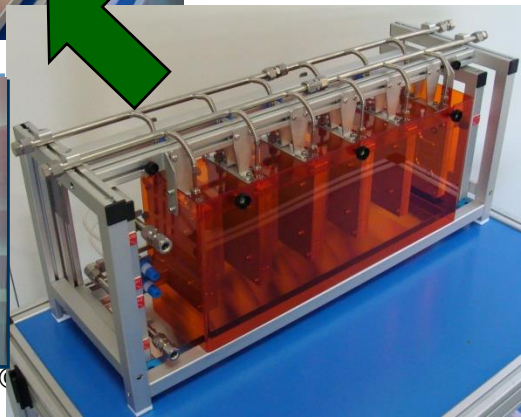
Corning Launched AFR™ G1 Photo Reactor

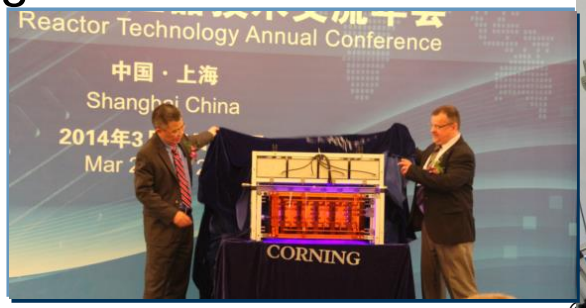
- An engineered reactor with all of the benefits from the existing G1 reactor (good mixing, good temperature management, etc)
- Plus:
  - Good control of the light source
    - Position of the LED guarantees homogenous illumination
    - LED temperature control guarantees long lifetime
  - Double illumination guarantees a more efficient use of the light
  - Choice of wavelength
    - 365 nm, 405 nm, etc.
  - Light intensity is tunable





LED Lighting Device





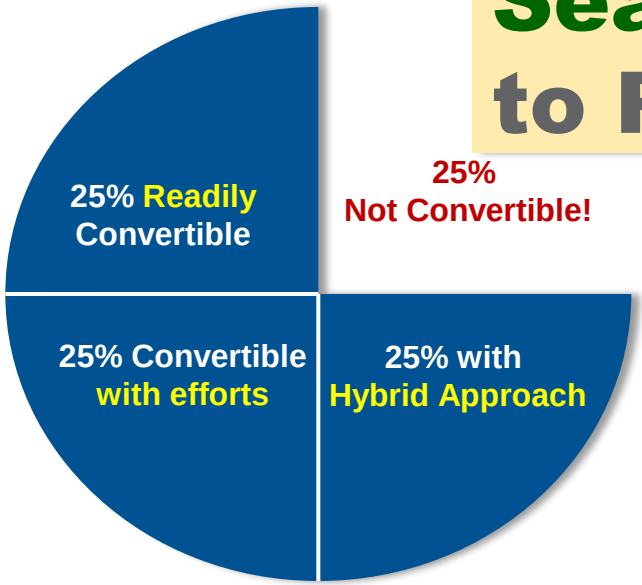
AFR: Revolutionary Improvement vs. Batch

1000 X HE Improvement

100 X Mixing Improvement

1/1000 X Batch Reactor Volume

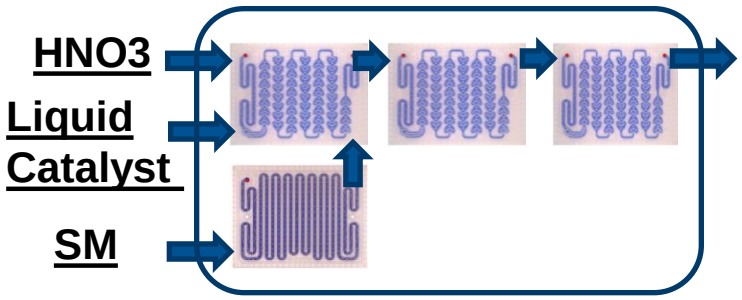
Seamless Scaleup from Lab to Production



- Not 100% reactions fit to AFR
- Continue requiring innovative process development
- Chemists, ChE, MechE work together

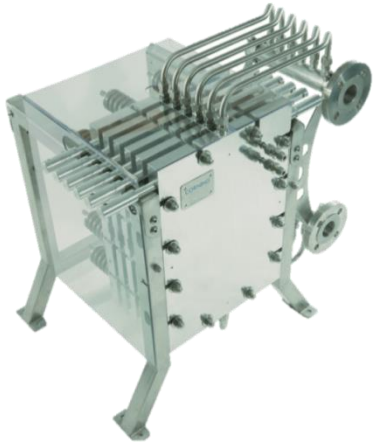
Continuous production of fine chemicals is real with Corning® AFR™: *Fuel Additive Production*

Process Development Done in G1
80 ton/yr throughput



Absolutely Seamless Scaleup

Production Rrun in G4
>2000 ton/yr throughput



- ✓ Process development was done on AFR™ G1 platform
- ✓ Optimized mole ratio, reaction temperature, flow rate, residence time, dosing method, and other parameters.
- ✓ Project duration: 4 weeks!

- ✓ Based on G1 data, Corning designed G4 reactor.
- ✓ All parameters are the same except the flow throughput increased by 25 times and led to annual throughput of 2000 tons.
- ✓ Absolutely seamless scale-up!

Continuous production of fine chemicals is real with Corning® AFR™: *Seamless scale-up from G1 to G4*

Seamless Scale up from G1 to G4 : Significantly changes equipment layout and safety management

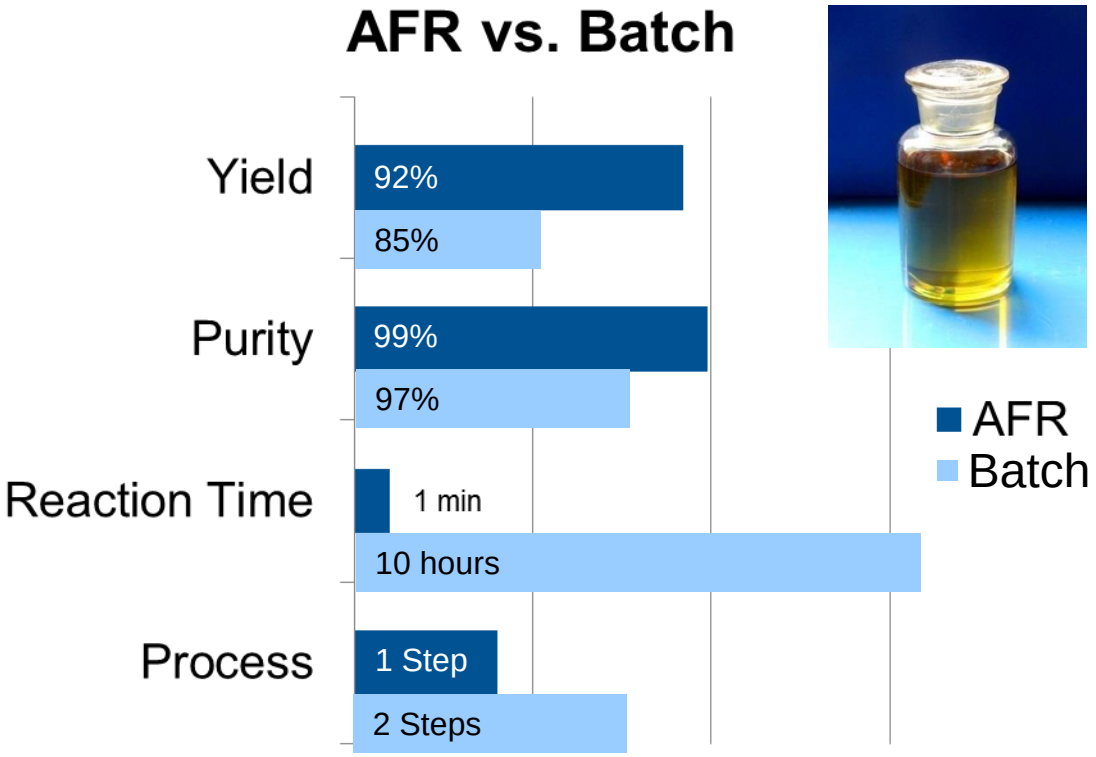
AFR Value Prop:

- ❑ **Inherently Safer Design**
- ❑ **Scaled flow up by >25 times from G1 to G4**
- ❑ **1st sample fully met product specs (2014.1)**
- ❑ **Identical yield (99.8%) achieved in G1 and G4**
- ❑ **Manpower reduced 70%**
- ❑ **Capex reduced by 30%**
- ❑ **Opex reduced by >30%**

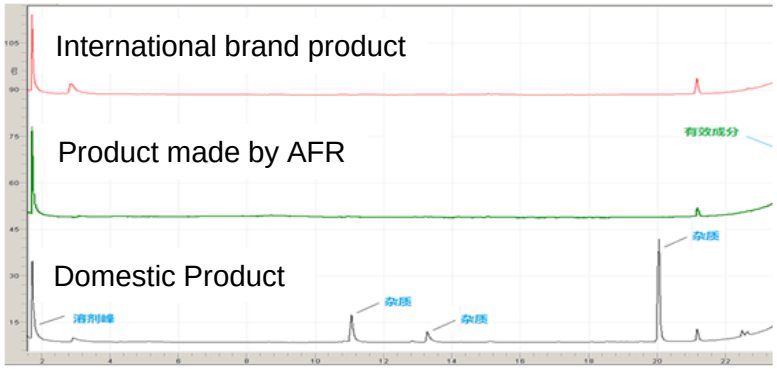
AFR enabled continuous synthesis of lubricant additives

-Simplified process with improved yield, purity, and efficiency

- "*Absolutely revolutionary process innovation!*" –*Chinese Media*
- Lubricant additives: **Y2010A *4,4'-Methylene bis(dibutyldithiocarbamate)* Cas No.:10254-57-6**
- AFR converted "2 step" complicated batch synthesis into "1 step" stable continuous synthesis
- Increased the yield from 85% to 92%
- Improved the purity from 97% to 99%
- Reduced reaction time from 10 hours to 1 minute
- Filed 3 process Chinese patents



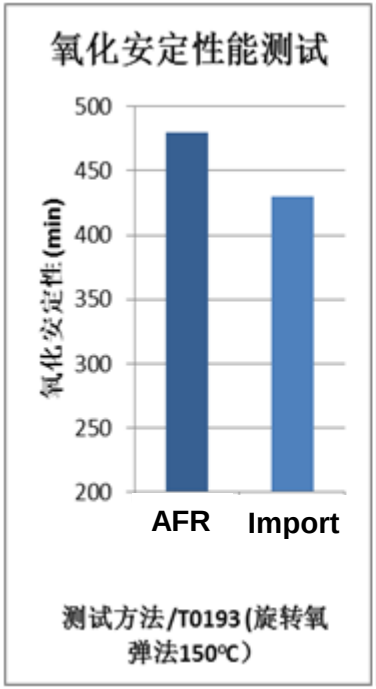
中国石化 SINOPEC



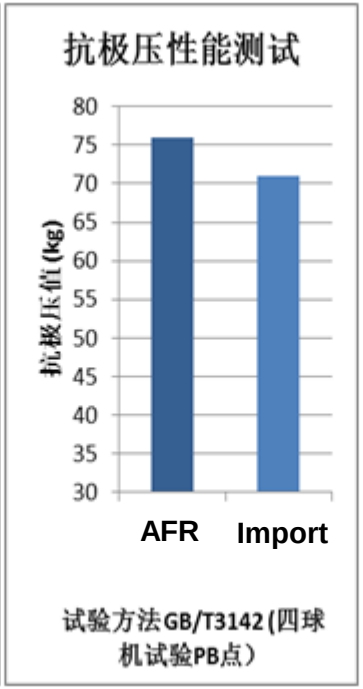
AFR enabled continuous synthesis of lubricant additives
- Y2010A application performance

中国石化 SINOPEC

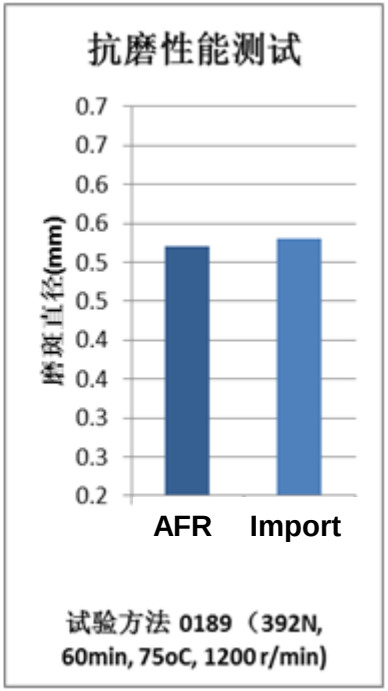
Oxidation Stability



Resistance on extreme pressure



Abrasive resistance



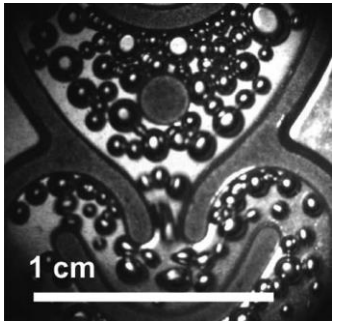
Application Performance Comparison with Import Brand Product

Selective Hydrogenation of Slurry Catalyst

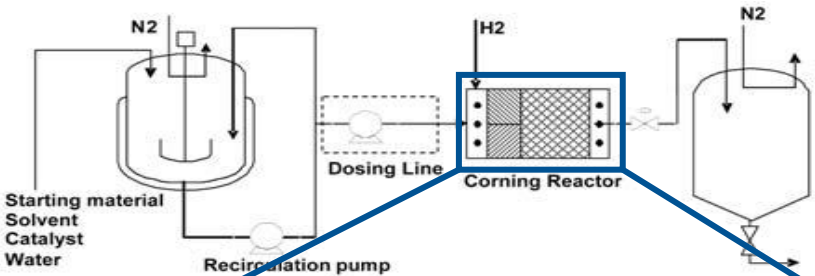
98%+ conversion & selectivity (impurity profiles within spec)

- **highly exothermic (>400 kJ/mol)**
- **30 µm catalyst in slurry**
- **significant catalyst reduction**

Ref: Chemistry Today • Vol 27 n 6 / November-December 2009



Excellent G/L Mixing



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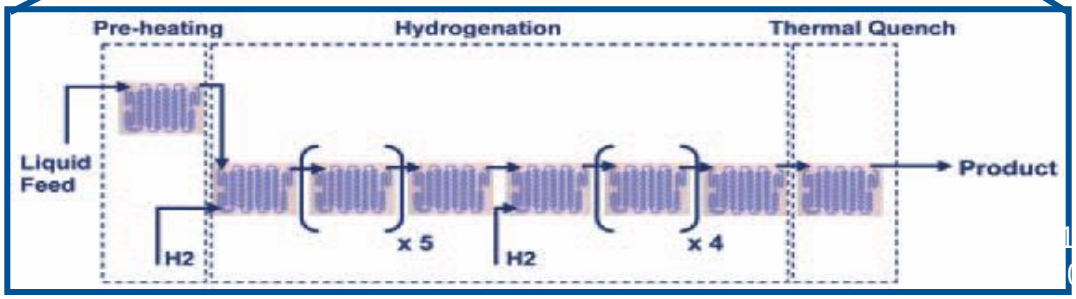
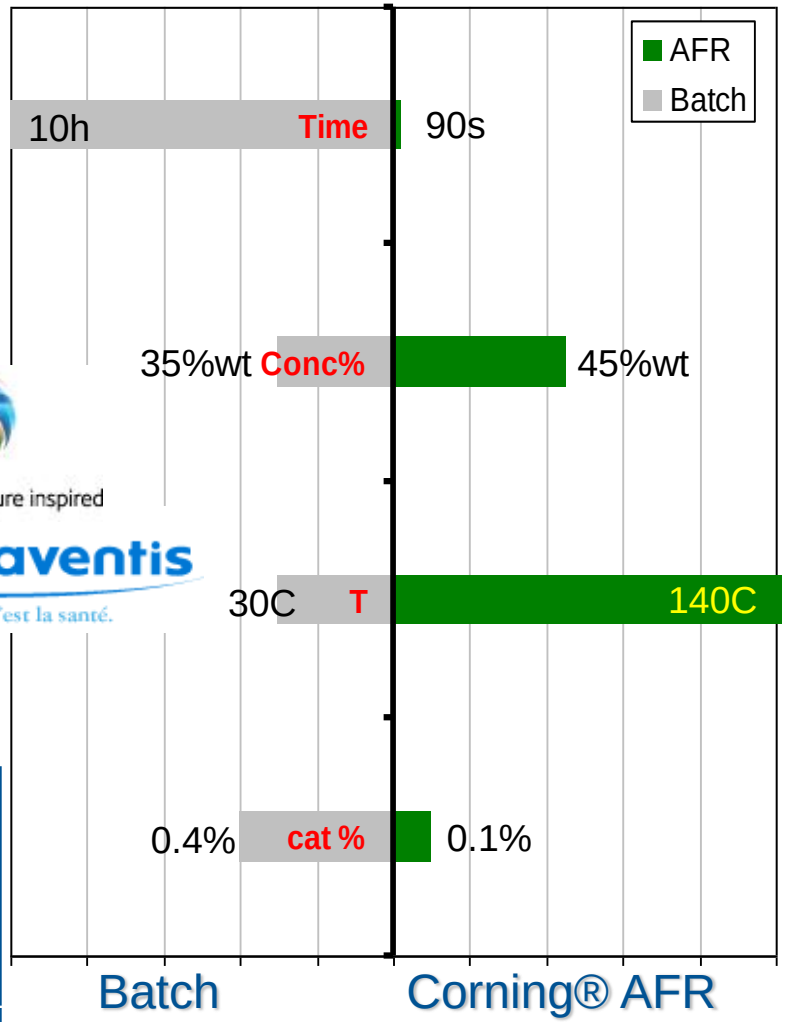
L'essentiel c'est la santé.

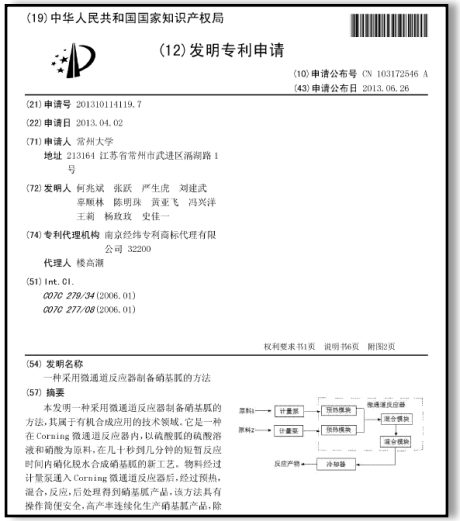




Changzhou University: China National Award for development of flow process of nitro-guanidine with AFR

- AFR Inherently Safer Design (ISD) enables tough chemical synthesis
- Undergrads team successfully developed continuous synthesis of a hazardous chemicals using Corning® AFR (2013 November)
- Developed AFR process won national highest award
- Developed AFR process package sold to Industry for >1.6M USD

Chinese Patent



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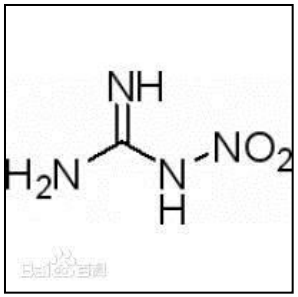
权利要求书1页 说明书6页 附图2页

(54)发明名称

一种采用微通道反应器制备硝基胍的方法

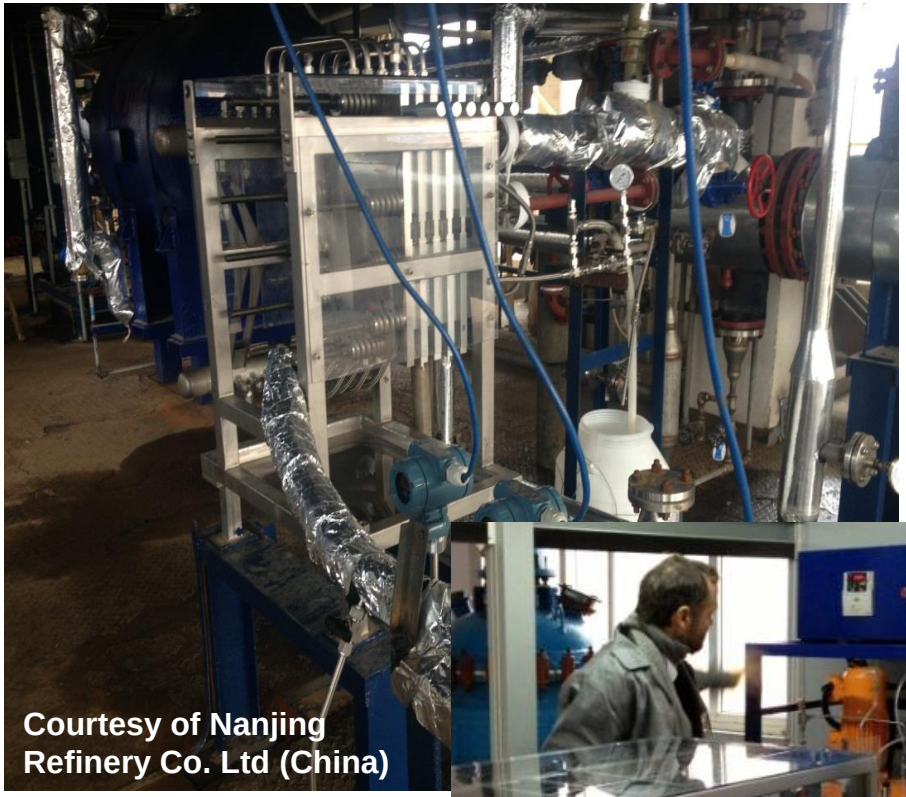
(57)摘要

本发明一种采用微通道反应器制备硝基胍的方法，其属于有机合成应用的技术领域。它是一种在Corning微通道反应器内，以硫酸胍的硫酸溶液和硝酸为原料，在几十秒到几分钟的短暂反应时间内硝化脱水合成硝基胍的新工艺。物料经过计量泵送入Corning微通道反应器后，经过预热，混合，反应，后处理得到硝基胍产品，该方法具有操作简便安全，高产率连续化生产硝基胍产品，除



National Highest Award

Production Plant Installations

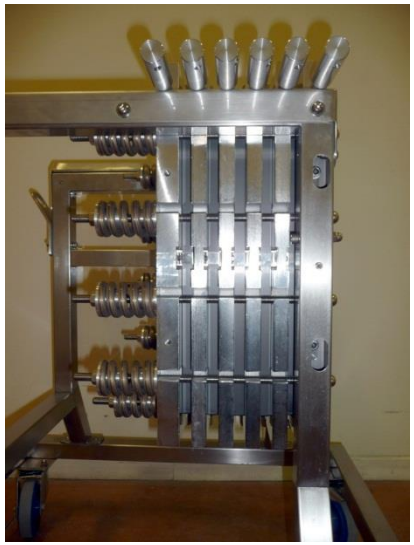


Courtesy of Nanjing Refinery Co. Ltd (China)

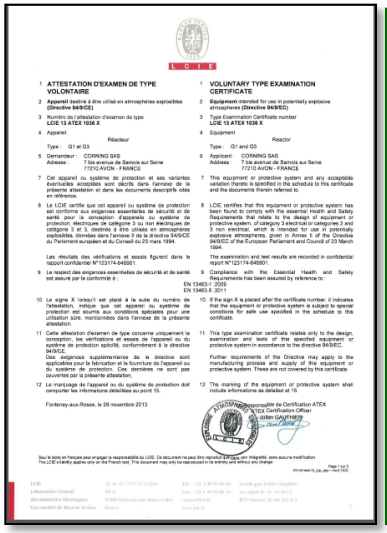
Courtesy of Shandong Brother Tech Co. Ltd (China)

Courtesy of Medichem (Spain)

AFR™ technology & manufacturing have received many certificates, and awards in Europe and Asia



Received 2013 National Innovation Award by China Petroleum & Chemical Industry Federation CPCIF



- **TÜV PED Quality Certificate (G4 SiC)**
- **China SELO A4 (SiC)**
- **EU ATEX II 2 G c IIC T4 Certificate (G1, G3) Certificates**
- **FDA Certificates for compliances**
- **ISO 14001**
- **…**

AFR helped Changzhou University win National Grand Award "Challenge Award"

2016 Corning Reactor Technology Global Conference
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2016 Corning Reactor Technology Global Conference Green Innovation Awards

Growing list of industry & academic AFR customers*

* Partial list

Growing list of industry & academic AFR customers*

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Flow Chemistry Process Development

Concluding Remarks

- *AFR™ technology is an Inherently Safer Technology (IST) that drastically reduces the consequences of an accident on the plant environment, however it does not prevent using the good & safe practices developed by the chemical industry.*

- *Successfully demonstrated seamless scale-ups providing customers increased confidence in adopting this technology*

- *We are proud of seeing our long-list of G4 production projects going on in Asia and Europe*

Contacts: *gosainp@corning.com*

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